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(54) AN INFRARED TECHNIQUE FOR MEASURING A PARAMETER, e.g. THICKNESS, OF A THIN FILM

(71) We, INFRA SYSTEMS, INC.,
 a corporation organised under the laws of the
 State of Ohio, United States of America, of
 650 Ackerman Road, Columbus, Ohio,
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 declare the invention, for which we pray that
 a Patent may be granted to us, and the method
 by which it is to be performed, to be particularly described in and by the following
 statement:—

This invention relates to an infrared technique for measuring a parameter e.g. thickness of a thin film applied to the surface of a supporting material.

In conventional techniques a beam of infrared radiation of a frequency which is partly absorbed by the material of the film is directed through the film, is specularly reflected off the surface of the supporting material, passes back through the film and is detected by a sensor. By comparing the intensity of the specularly reflected beam after passing through the film with the intensity of a reference beam of a frequency which is not substantially absorbed by the film a measure of the thickness of the film is obtained. For further details of such apparatus the reader is referred to U.S. Patent Specification No. 3017512.

A problem encountered in the practical application of such prior techniques is that a part of the beam reflected specularly from the top surface of the film interferes with the main part of the beam reflected specularly from the surface of the supporting material. The resulting interference pattern can lead to false readings.

According to the invention, there is provided a method of measuring a parameter (e.g. the thickness) of a thin film applied to the surface of a supporting material, in which two infrared beams of different wavelengths pass into the film, are diffusely reflected by the supporting material and then pass out of the film, and in which the intensity of a diffused part of one beam received by a sensor is compared with the intensity of a diffused part of the other beam received by the sensor to give a measure of the said parameter.

Also, according to the invention, there is provided apparatus for measuring a parameter (e.g. the thickness) of a thin film applied to the surface of a supporting material comprising: a source of radiation which, in operation, directs two infrared beams of different wavelengths towards the film on the surface of the supporting material; a sensor arrangement which is responsive to infrared radiation of the said wavelengths and is constructed and arranged to detect parts of the beams which have been diffusely reflected from the supporting material and to give two output signals each related to the intensity of one of the detected beam parts; and means for comparing the output signals to give a measure of the said parameter.

As will become apparent from the following description, it is possible in carrying out the present invention to arrange the sensor so that it receives only radiation reflected from the supporting material thereby eliminating interference from radiation reflected from the surface of the film.

The measure of the thickness of the film, which will normally be in the form of an electrical signal can be used to operate a visible indicator graduated appropriately or may be used to control machinery by which the film is spread onto the supporting material.

It will be apparent that the invention is most applicable to films which are applied to a relatively matt or rough supporting surface to increase the proportion of radiation which is reflected diffusely from it.

In the following description of this invention and the appended claims, the term diffusely reflected is intended to include radiation components diffusely reflected at the

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second surface of the film or diffusely reflected from the substrate, or both. Typical materials for the substrate include foils that may be opaque to infrared radiation or paper which may be dispersive of the radiation and thus effectively produce diffusely reflected radiation components from the radiation thus scattered. An organic material such as polyethylene often forms the film in the commercially available products and when such a material is formed as a coating on a diffuse surfaced substrate, the resultant film will have an exposed or outer surface (referred to as the first surface hereinafter in this specification) which is specular and an opposite or interface surface (referred to as the second surface hereinafter in this specification) which is diffuse. In the case of a substrate formed from a dispersive material, the substrate interface surface may also diffusely reflect incident radiation in addition to diffuse reflection from the substrate body.

The invention will now be particularly described by way of example with reference to the accompanying drawings of a measurement apparatus constructed in accordance with the invention.

Referring now to the drawing, an apparatus of the dual-beam, infrared radiation reflection type utilizing the interference elimination technique of this invention is diagrammatically illustrated with respect to a double-layer sheet-form material comprising a base sheet or substrate B having a film or coating C applied to a surface of the substrate. In this illustrative example, it is assumed that the substrate B and the coating C are a portion of an elongated sheet-form web shown in vertical section and which has been formed by a process wherein the film or a material forming the film was applied to a surface of the substrate and is bonded thereto forming a unitary structure. It is the objective of the illustrated apparatus to determine a parameter of the film C which, for example, may be the film thickness d . The ultimate objective of determination of a particular film parameter, such as the thickness, may be to provide means of effecting control over a continuous process, as for example, control over the thickness d of the film C as it is applied to the substrate C. In the material to which the measurement technique of this invention is applicable, the substrate B is formed from a material such as paper or a foil which may be opaque to infrared radiation and has a diffuse surface D at the interface with the film C producing diffuse reflection of radiation or which, in the case of a radiation-scattering substrate, may also produce back-scattering or simulated diffuse reflection in the substrate. The film C is formed from a material transmissive of the infrared radiation, an organic polyethylene

material, for example, and has a specular outer surface S.

Apparatus for generating the two beams of infrared radiation and subsequently detecting the reflected components of each beam is only diagrammatically illustrated in the drawing as the several components comprise structures well-known in the art and includes a dual-beam, infrared radiation source designated generally by the numeral 10, a radiation responsive sensor 11 and a signal amplification and analysis circuit 12. The radiation source 10 is illustrated as being of the type which is capable of generating two discrete beams of infrared radiation having the respective wavelengths λ_1 and λ_2 , and directing the two beams in angularly incident relationship toward the specular surface S of the film C for reflection at both the specular surface and the diffuse surface D or scattering from dispersive type substrate material. In this example, the two beams of radiation are illustrated as being directed along the same path and the radiation source is of the type that generates the two beams in time-spaced relationship for transmission to the film in an alternating fashion. The illustrated beam-path is applicable to both beams for the purpose of illustrating the technique of this invention for eliminating interference error effects of the optical interference phenomenon. U.S. Patent No. 3,089,382 is illustrative of the type for dual-beam radiation generating apparatus which may be utilized for producing the two-time-spaced beams of radiation for the reflective-type measurement technique such as is basically disclosed in the previously mentioned U.S. Patent No. 3,017,512. In this type of apparatus, an infrared radiation source producing polychromatic radiation is arranged to direct the emitted radiation toward the subject under test and the radiation is caused to pass through filter elements which are alternatively interposed in the path of the radiation to form the two beams of radiation of different wavelengths. These filter elements, which are of the band-pass-type having a designed pass-band of a desired wavelength spectrum, are mechanically supported in the path of the radiation by means such as a revolving wheel to alternately pass through the path of the radiation and produce two time-separated beams of monochromatic radiation. The wavelength of the designed pass-band for each filter element is determined by the specific film material and the property or parameter of interest in a particular test arrangement to provide on beam of radiation, the reference beam, of a wavelength λ_1 that, preferably, is not absorbed by the material while the other beam of radiation, the absorption beam, is of a wavelength λ_2 selected to exhibit a characteristic resonance absorption. It is not necessary that the reference beam be totally

unabsorbed by the film but only that its absorption be significantly less than that of the absorption beam. Accordingly, the two beams of radiation are transmitted unequally through the film due to absorption of one beam and this difference is detectable by an appropriate detection system to provide an output.

The radiation responsive sensor 11 is disposed in predetermined angular relationship to the source 10 so that only diffusely reflected radiation components will be incident on a radiation-receptor-surface of the sensor. Specific characteristics of the sensor 11 are determined by the particular radiation wavelengths utilized in a specific test arrangement and the sensor may be of the type known as a photovoltaic cell or it may be a photoresistive device. Either type of sensor includes terminals providing means of connection to an electronic amplifier circuit and the characteristics of the detector or sensor are utilized to form a signal at the output of the amplifier. This amplified output signal is subsequently fed to respective amplifier circuits 14 and 15 that respond to signal components related to the two specific radiation beams. An output signal from each amplifier circuit 14 and 15 is then fed into a ratio-analyzer-circuit 16 and the output of the ratio circuit subsequently drives a readout device 17 such as in indicating meter. Alternatively, although not shown, the output signal from the ratio-analyzer-circuit 16 may be utilized to drive a process control circuit. Control over alternate routing of the signal from the amplifier 13 to the respective amplifier circuits 14 and 15 is controlled by an electronic switching circuit 18 which functions in timed relationship to the operation of the source 10 in generating the two discrete beams of radiation. For example, the mechanisms controlling operation of the filtering mechanism can be utilized to provide a signal to synchronously operate the switching circuit 18. A circuit connection for this switching signal is indicated graphically at 19. In view of the previous detailed disclosures of radiation generation and detection apparatus as in Patent 3,089,382, it is believed that the foregoing brief description of apparatus components will be adequate for a complete understanding of the interference eliminating technique of this invention.

Both beams of radiation travel along the same path and are incident to the specular, first surface S of the film C and a portion of the radiation will be specularly reflected. The path of incidence is designated by the letter I in the drawing and the path taken by that portion of the radiation specularly reflected at the first surface S is designated by the letter R_s. Angles of incidence θ_i and reflection θ_r with respect to a normal to the surface S at the point of incidence are also

designated in the drawing. Not all radiation incident to the specular surface S will be reflected at that surface and portions of each beam of radiation enter the film C and are incident on the diffuse second surface D. While the film C normally comprises a material having an index of refraction different from that of the intervening space between the source 10 and the film, the effects of any resultant refraction have not been illustrated in the drawing and are not discussed in the specification as both beams are affected to substantially the same extent and the effect is self-cancelling since each beam exits the film into the same space. Those portions of each beam incident to the second surface D will be diffusely reflected resulting in a multiplicity of radiation components indicated generally at R_d directed toward the surface S in relatively divergent relationship. The diffusely reflected radiation may include a component R_{ds} which is aligned with an assumed path of reflection that is at angle of reflection equal to the angle of incidence and thus parallel to the path of the specularly reflected radiation R_s; however, a substantial amount of the diffusely reflected radiation will be directed along paths oriented at angles other than that of the specular reflection and angle θ_r . A relatively large proportion of the diffusely reflected radiation will be directed along paths which are oriented at angles greater than the specular reflection angle θ_r . Several paths for diffusely reflected radiation are shown in the drawing for the purpose of illustrating the effect but are not intended to be indicative of an actual pattern of reflected radiation or the magnitude of the reflected radiation component at any specific angle.

As previously indicated, the radiation-responsive sensor 11 is geometrically oriented relative to the reflected radiation to prevent incidence of specularly reflected radiation R_s at the radiation receptor surface of the sensor. This technique limits the detected radiation to only those radiation components which are diffusely reflected from the substrate B and, as shown in the drawing, the sensor is positioned to further limit detection of diffusely reflected components to those that are reflected at an angle which is greater than the angle of reflection θ_r for specularly reflected radiation. Detection of only the diffusely reflected components of each beam prevents introduction of measurement errors due to interference between beam components that are relatively phase displaced as are the first surface specularly reflected components and the diffusely reflected components. This is accomplished by positioning the radiation-responsive sensor 11 in angularly spaced relationship to the radiation source 10 such that the receptor surface of the sensor will not extend into the space through which

the specularly reflected components R_s pass. The specific angular relationship in any particular test arrangement is determined by the physical size of the sensor's receptor surface and the diffuse reflection pattern to obtain a maximum response. It will be noted that the drawing does not accurately illustrate the proportional geometric relationship of the several radiation components as the specularly reflected components R_s and R_{ss} which are spaced parallel at a distance determined by the thickness of the film C and would be relatively close together in most test applications since the usual films are relatively thin. In view of this practical aspect of measurement of these films, the detector 11 would be positioned so that no diffusely reflected components at a reflection angle less than θ_r would be incident on the detector. While the drawing is only two-dimensional, it will be recognized that the diffusely reflected radiation will have a third dimension but this does not affect illustration of the technique of positioning the sensor 11 to only be responsive to diffusely reflected radiation.

The techniques of this invention are specifically applicable to measurement of a parameter of a film C formed from an organic material such as polyethylene. Measurement results of a polyethylene film parameter are affected by the specific wavelengths λ_1 and λ_2 selected for the respective reference and absorption beams and it has been found that the thickness of the film C also is a factor in determining the wavelengths for optimum results. For thick polyethylene films which exceed 0.0005 inches in thickness, up to about 0.010 inch, the preferred absorption wavelength is selected to be within the 2.30—2.60 micron wavelength spectrum and the reference wavelength which does not exhibit absorption is preferably selected to be relatively close to the absorption wavelength and, in this instance, is 2.25 ± 0.02 micron. For relatively thin polyethylene films, those films having a thickness dimension less than 0.001 inch, the optimum wavelength of radiation exhibiting absorption is 3.45 ± 0.02 micron, and the reference wavelength which is preferably selected to be close is either 2.65 ± 0.02 micron or 3.75 ± 0.02 micron.

The foregoing detailed description is specifically directed to the illustrative example shown in the drawing; however, as previously stated, this measurement technique is also applicable to the case where the substrate is transmissive of the infrared radiation to a degree but is dispersive. In connection with measurements of film formed on a dispersive substrate, it will be readily seen that there would be radiation components diffusely scattered or reflected at numerous levels or depths within the substrate in a manner simi-

lar to the illustrated diffuse reflection at the interface surface. Suitable orientation of the radiation sensor will in this case also eliminate interference from specularly reflected components of the beams thereby avoiding interference errors in the same manner as previously described. It is necessary in the case of scattered radiation to eliminate any error arising from absorption of either or both the reference and absorption beams by the substrate material. This can be accomplished best by selecting wavelengths for each beam which do not exhibit absorption in the substrate material. If selection on this basis is not possible, then selection of wavelengths for each beam to exhibit equal absorption in the substrate or to select wavelengths such that the ratio of absorption for each beam is substantially different for the film and the substrate.

This measurement technique for eliminating interference-caused errors in infrared measurement is particularly advantageous with respect to films or coatings where the second reflective surface is diffuse or where the substrate comprises a dispersive material producing diffuse scattering (effectively diffuse reflection for the purposes of this measurement technique) and is a relatively ineffective technique when applied to films having a relatively smooth or specular second surface. Performing reflective measurements to utilize only diffusely reflected or diffusely back-scattered components avoids error introduced by interference from a relatively phase displaced beam of radiation or components of such a beam. Positioning of the radiation sensor in angularly spaced relationship to the incident beams of radiation to prevent specularly reflected radiation from being incident to the radiation-receptor surface of the sensor accomplishes the objective of eliminating interference errors. Selection of appropriate wavelengths for the absorption and reference beams further enhances the accuracy of the reflection measurement with respect to a polyethylene material.

WHAT WE CLAIM IS:—

1. A method of measuring a parameter (e.g. the thickness) of a thin film applied to the surface of a supporting material in which two infrared beams of different wavelengths pass into the film, are diffusely reflected by the supporting material and then pass out of the film, and in which the intensity of a diffused part of one beam received by a sensor is compared with the intensity of a diffused part of the other beam received by the sensor to give a measure of the said parameter.

2. A method according to Claim 1 in which the said diffused parts of the beams are reflected at angles greater than parts

specularly reflected from the interface between the film and the supporting material.

5 3. A method according to Claim 1 or 2 for measuring a polyethylene film having a thickness greater than 0.0005 inch in which one beam which is most absorbed has a wavelength in the range of 2.30 to 2.60 micron, the other beam having a relatively short wavelength.

10 4. A method according to Claim 2 in which the beam of shorter wavelength has a wavelength of 2.25 plus or minus 0.02 micron.

15 5. A method according to Claim 1 or 2 for measuring polyethylene film having a thickness less than 0.001 inch, in which the beam which is most absorbed has a wavelength of 3.45 plus or minus 0.02 micron and the other beam has a relatively short wavelength.

20 6. A method according to Claim 3 in which the beam of shorter wavelength has a wavelength of 2.65 plus or minus 0.02 micron or 3.75 plus or minus 0.02 micron.

25 7. A method according to claim 1 and substantially as described with reference to the accompanying drawings.

30 8. Apparatus for measuring a parameter (e.g. the thickness) of a thin film applied to the surface of a supporting material comprising: a source of radiation which in operation directs two infrared beams of different wavelength towards the film on the surface of the supporting material; a sensor arrangement which is responsive to infrared radiation of the said wavelengths and is constructed and arranged to detect parts of the beams which have been diffusely reflected from the supporting material and to give two output signals each related to the intensity of one

of the detected beam parts; and means for comparing the output signals to give a measure of the said parameter. 40

9. Apparatus according to Claim 8 in which the sensor arrangement is arranged to receive only radiation reflected at angles greater than parts reflected specularly from the interface between the film and the supporting material. 45

10. Apparatus according to Claim 8 or 9 for measuring the thickness of a polyethylene film in which the source of radiation is constructed to produce a first beam at a wavelength within the range of 2.30 to 2.60 micron. 50

11. Apparatus according to Claim 10 in which the source of radiation is constructed to produce the second beam at a wavelength of 2.25 plus or minus 0.02 micron. 55

12. Apparatus according to Claim 8 or 9 for measuring the thickness of polyethylene film in which the source of radiation is constructed to produce a first beam at a wavelength of 3.45 plus or minus 0.02 micron. 60

13. Apparatus according to Claim 12 in which the source of radiation is constructed to produce the second beam at a wavelength of 2.65 plus or minus 0.02 micron or 3.75 plus or minus 0.02 micron. 65

14. Apparatus substantially as herein described with reference to the accompanying drawings. 70

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